Minerals Engineering 92 (2016) 160–167

Contents lists available at ScienceDirect

Minerals Engineering

journal homepage: www.elsevier.com/locate/mineng

Modification of ilmenite surface properties by superficial dissolution method

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ARTICLE INFO

Article history:
Received 31 December 2015
Accepted 12 March 2016
Available online 18 March 2016

Keywords:
Ilmenite
Flotation
Acid surface dissolution
Surface properties
Zeta potential

ABSTRACT

Acid surface dissolution as a pretreatment method converts Fe^{2+} ions on the ilmenite surface to Fe^{3+} ions. XPS analysis showed that the content of Fe^{3+} increases from 48.5% to 59.8% after surface dissolution for 15 min in a solution of sulfuric acid with a concentration of 10%. This conversion, without any phase transformation, decreases the zeta potential of ilmenite in a wide pH range, resulting in a shift in IEP (Iso-Electric Point) from a pH of 5.4 to 2.3. FTIR spectra and zeta potential measurements showed that the increase of oleate ions adsorption on the ilmenite surface, resulting from the surface dissolution process, is insignificant. After surface dissolution, the formation of more ferric iron oleate species ($Ksp = 10^{-29.7}$) being more stable than ferrous iron oleate ($Ksp = 10^{-15.5}$) compounds yields an increase of ilmenite hydrophobicity and floatability in a wide pH range. Using 3.65×10^{-4} M sodium oleate at a pH of 6.3, the maximum flotation recoveries are obtained as 73.5% and 92% for non-treated and acid pretreated ilmenite, respectively.

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1. Introduction

Titanium has many applications due to its high strength-to-weight ratio and inertness in many corrosive environments. Ilmenite and rutile are the most important and economical titanium minerals (Bulatovic and Wyslouzil, 1999; Chachula and Liu, 2003). Nowadays, because of the decreasing availability of natural resources of rutile, ilmenite has become the most important mineral for the production of titanium metal and titanium dioxide (Samal et al., 2009). The detailed formula of ilmenite as a titanate of ferrous iron mineral ($Fe^{2+}Ti^{4+}O_3$) is (Fe, Mg, Mn)TiO_3, with only a limited amount of Mg and Mn substituted for Fe or Ti in its lattice (Deer et al., 1991; Didenko and Efremov, 2004; Pownceby et al., 2008). The gravity, magnetic and electrostatic separation methods or their combinations are the conventional methods for preparing an ilmenite concentrate. However, these methods cannot be used efficiently for processing of ilmenite when it is finely disseminated within the gangue minerals. In these cases, the froth flotation is suggested as an effective technique for ilmenite concentration (Song and Tsai, 1989; Fan et al., 2009; Fan and Rowson, 2000a,b). Ilmenite shows poor floatability in comparison with other oxide minerals such as magnetite and rutile, even after using large amounts of collector. This is due to the interaction of just half of the metallic cations (Fe^{2+} and Ti^{4+}) as the surface active sites with the collector anions in each pH range (Fan and Rowson, 2000a,b; Fan et al., 2009). Several methods such as microwave irradiation (Fan and Rowson, 2000a; Fan et al., 2000; Fan and Rowson, 2002; Fan et al., 2009; Irannajad et al., 2014; Nuri et al., 2014), oxidation roasting (Mehdilo et al., 2014), surface activation (Fan et al., 2000), hot flotation (Liimatainen and Techn, 1977; Gutierrez, 1976), agglomeration flotation (Runolinna and Rinne, 1960) and reverse flotation (Behera and Mohanty, 1986) have been used to modify ilmenite surface properties, as well as improving its floatability.

The surface dissolution is another method which has been used for modification of minerals surfaces and creation of differences in their flotation behavior (Abeidu, 1974; Abeidu and Kholeif, 1976; Zhong and Cui, 1987; Torem et al., 1992; Scorzelli et al., 1996; Bulatovic and Wyslouzil, 1999; Moon and Fuerstenau, 2003; Liu et al., 2006; Wang and Yu, 2007; Zhu et al., 2011; Feng et al., 2012). Surface dissolution changes the distribution and states of elements on a mineral surface which affects the surface properties of minerals. This pretreatment method also changes the solution chemistry of the flotation system by various ions entering the liquid (Wang and Hu, 1988). Ananthapadrnanabhan and Somasundaran (1981) indicated that the extent of surface adsorption and rate of adsorption are influenced by the nature of the cation due to oleate adsorption at the cationic sites of the minerals. The superficial leaching of beryl by cold HCl and the mixture of HCl and HF has improved its floatability through dissolving some

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http://dx.doi.org/10.1016/j.mineng.2016.03.016
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cations (such as Si) from the surface (Torem et al., 1992). The selectivity of pyrochlore flotation from silicate minerals has been increased by surface dissolution in HCl solution at pH = 2 (Abeidu, 1974). Moon and Fuerstenau indicated that the flotation recovery of spodumene is enhanced when the mineral was treated in an alkaline solution (pH = 12) prior to flotation with oleate at a solution pH of 8. In their research, the improvement in spodumene floatability was not observed after surface dissolution in an acidic solution (Moon and Fuerstenau, 2003). Wang and Yu showed that the floatability of spodumene and beryl is increased after surface dissolution in an alkaline solution (Wang and Yu, 2007). Scorzelli et al. (1996) reported that the superficial leaching pretreatment with HCl, HNO_3 and HF changes the surface properties of xenotime and zircon, and enhances their flotation recoveries. Acid surface dissolution pretreatment has also been used for improvement of ilmenite and perovskite flotation behavior (Abeidu and Kholeif, 1976; Zhong and Cui, 1987; Bulatovic and Wyslouzil, 1999; Zhu et al., 2011). Zhu et al. (2011) showed that the acid surface dissolution increases the ilmenite floatability and also improves its selective separation from titanaugite by the froth flotation method.

The aim of this study is to investigate the effect of acid surface dissolution on the physio-chemical properties of ilmenite, including its floatability, surface oxidation, zeta potential, collector adsorption and finally its morphological features.

2. Materials and methods

2.1. Materials

The purified ilmenite sample was prepared from Qara-aghaj hard rock deposit located in the north-west of Iran. The hand-picked samples, taken from the deposit, were crushed and ground to less than 150 μm, a size which has been determined as the ilmenite liberation degree (Mehdilo and Irannajad, 2010). Then, the purification process was conducted in order to prepare pure ilmenite by sieving and several stages of tabling, with low and high intensity magnetic separation methods. The purified sample was washed several times with distilled water and dried at room temperature. Examination under a binocular microscope showed that the ilmenite grains had clean surfaces and were free of gangue minerals. The chemical composition of the purified ilmenite is presented in Table 1. The important impurities in the sample are MgO (2.53%) and MnO (1.04%). X-ray diffraction (XRD) analysis showed the ilmenite as the primary mineral phase in the sample. Thus, the impurities are related to the substitution of Mg and Mn for some Fe^{2+} ions in the ilmenite structure.

Sodium oleate ($CH_3(CH_2)_7CH{=}CH(CH_2)_7COONa$) with 95% purity owned by Sigma Aldrich was used as a collector. The analytical grade H_2SO_4 (97%) and NaOH (98%) were used for pH adjustment and distilled water was used in all the experiments. H_2SO_4 was also used in the acid surface dissolution process.

2.2. Methods

2.2.1. Material characterization

X-ray fluorescence (Philips X Unique II) was used in order to determine the chemical composition of the ilmenite sample. The XRD pattern of the ilmenite samples was prepared using XPERT MPD diffractometer with Cu Ka radiation. The comparisons of textural and morphological features of ilmenite samples before and after acid surface dissolution were performed using Philips XL30 model Scanning Electron Microscopy (SEM).

2.2.2. Surface dissolution

The surface dissolution pretreatment on the ilmenite sample was performed by mechanical stirring in a 10% sulfuric acid solution for 15 min. After surface dissolution, the liquid phase was poured out and the pretreated solid particles (6 g) were placed in a beaker, which was then filled with distilled water (50 mL). The suspension was agitated and washed with mechanical stirring for 4 min. Subsequently, the liquid was discarded and the remaining solid was filtered and dried at room temperature.

2.2.3. Flotation experiments

The flotation experiments were conducted on ilmenite and pretreated ilmenite in a 300 cm^3 Hallimond tube. In each experiment, the mineral suspension was prepared by adding 2 g of ilmenite (before and after surface dissolution) with a size of −0.15 +0.045 mm to the distilled water and conditioned for 5 min. Then, the collector was added to the suspension and conditioned for 8 min during which the pH was adjusted to the required value. The prepared pulp was then transferred to the Hallimond tube and flotation was carried out for 4 min. After the flotation tests, the sunk and floated products were filtered, dried, and weighed for calculating the flotation recovery.

2.2.4. XPS

The XPS analysis was carried out using a Specs EA 10 X-ray photoelectron spectroscope on the ilmenite surface, before and after acid surface dissolution pretreatment. This analysis was performed to determine the relative content of Fe^{2+} and Fe^{3+} ions on the surface of ilmenite. An Al K_α X-ray source at 14,866 eV was used in the XPS analysis. The measurements were carried out inside the analysis chamber, operating in a high vacuum of about 10^{-7} Pa. Binding energies were calibrated using characteristic carbon (C1s = 284.7 eV). In the XPS analysis, similar to the flotation experiments, the samples with a size of −150+45 μm were used.

2.2.5. FTIR

The FT-IR analyses were performed by NEXU670 FT-IR (Nicolet Corporation, USA). The conditioning of the suspension was carried out on the mineral sample ground to less than 0.015 mm, similar to the flotation experiments. In the quantitative analysis, the ratio of KBr to the sample was 300:1; wt/wt.

2.2.6. Zeta potential

The zeta potential measurements before and after acid surface dissolution were conducted using a Malvern instrument (UK). In each test, the preparation of suspension was carried out by adding 50 mg of pure ilmenite with a size less than 0.015 mm to 100 mL of distilled, deionized water containing 10^{-3} mole/L KCl as a supporting electrolyte. The prepared suspension was conditioned for 15 min during which suspension pH was measured. Zeta potential was measured by following the procedures described in the instrument's manual. The reported results are the average of at least three fully repeated experiments. The repeated tests showed a measurement error of ±2 mV.

Table 1
Chemical composition of the purified ilmenite.

Composition	TiO_2	Fe_2O_3	MnO	V_2O_5	P_2O_5	CaO	MgO	SiO_2	Al_2O_3	Co_3O_4	S	L.O.I
wt%	46.2	48.6	1.04	0.29	0.24	0.38	2.53	0.19	0.44	0.025	0.024	0.0

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